भारतीय मानक
Indian Standard

IS 8702 : 2015
(Reaffirmed 2020)

डाईयूरान, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Diuron, Technical — Specification

(First Revision)

ICS 65.100.20

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Pesticides and Pesticides Residue Analysis Sectional Committee, FAD 01

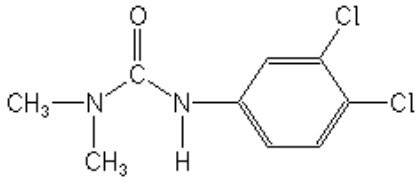
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides and Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Diuron, technical is used in the preparation of herbicidal formulations for agricultural use. It is also used selectively in certain crops.

Diuron is the accepted common name by the International Organization for Standardization (ISO) for 3-(3, 4-dichlorophenyl)-1, 1-dimethylurea.

The empirical and structural formulae and the molecular mass of diuron are indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_9H_{10}Cl_2N_2O$		233.1

This standard was first published in 1978. This standard has been revised to incorporate:

a) HPLC method for determination of diuron content in addition to volumetric method,
b) Amendment No. 1, 2 and 3, and
c) Update referred standards.

In the preparation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DIURON, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Diuron, technical.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

Diuron, technical shall be in the form of white to off-white colour homogenous powder, free from extraneous impurities or added modifying agents.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in clean and dry, mild steel drums, with a polyethylene liner of thickness not less than 0.062 mm. The containers shall also meet the general requirements given in IS 8190 (Part1).

5 MARKING

5.1 The containers shall be securely closed and the following information shall be marked legibly and indelibly on each container in addition to any other information, as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal diuron content, percent (*m/m*);
h) Cautionary notice worded as in the *Insecticides Act*, 1968 and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules,* 2011.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*,

Table 1 Requirements for Diuron, Technical

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Cl of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Diuron content, percent by mass, *Min*	95.0	A	—
ii)	Melting point, °C	158-159		**6**
iii)	Moisture content percent by mass, *Max*	0.07	—	**4.1**
iv)	Material insoluble in acetone, percent by mass, *Max*	Nil	—	**9**
v)	Acidity (as H_2SO_4), percent by mass, *Max*	0.12	—	**11.3.2**

1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 10946.

7 TEST

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF DIURON CONTENT

A-0 GENERAL

A-0.1 Any of the three methods, namely, aliphatic amine method (*see* **A-1**), aromatic amine method (*see* **A-2**) and HPLC method (*see* **A-3**) shall be used for the determination of diuron content. However, in case of a dispute HPLC method shall be the referee method.

A-1 ALIPHATIC AMINE METHOD

A-1.1 Apparatus

A-1.1.1 *Hydrolysis Apparatus* — *See* Fig. 1.

A-1.2 Reagents

A-1.2.1 *Standard Sulphuric Acid* — 0.1 N.

A-1.2.2 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-1.2.3 *Glycerol*

A-1.2.4 *Potassium Hydroxide Solution* — 20 percent, aqueous solution.

A-1.2.5 *Silicone Defoamer*

A-1.3 Procedure

A-1.3.1 Assemble the hydrolysis apparatus. Add 50 ml of standard sulphuric acid, accurately measured from a burette, and 50 ml of methanol to the condensate beaker. Transfer an accurately weighed portion of the sample, containing 0.5 to 0.6 g of active ingredient, into the round bottom flask. Add 15 ml of ethanol, and swirl to disperse the sample. Add 100 ml of glycerol, several drops (6-8) of silicone defoamer, one or two boiling chips and 100 ml of potassium hydroxide solution and immediately attach the reaction flask to the condenser *A*. With cooling water flowing through

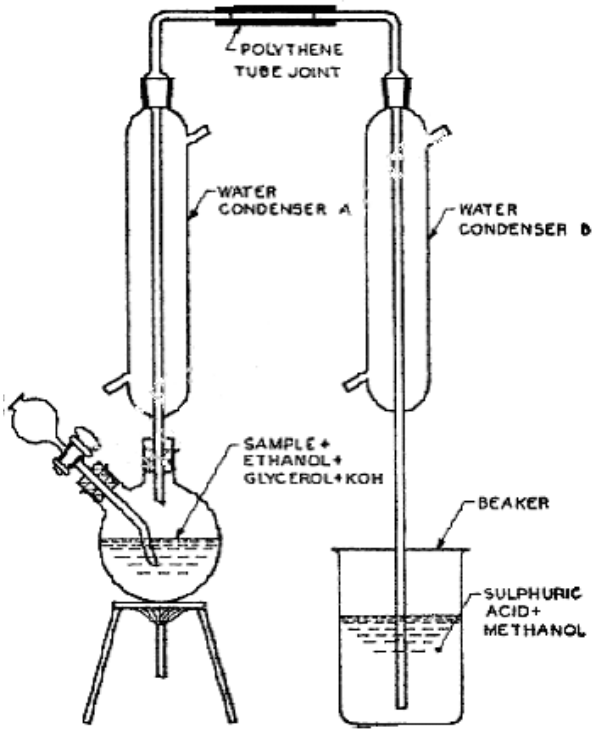


FIG. 1 HYDROLYSIS APPARATUS FOR DIURON CONTENT

each condenser independently, reflux the mixture for 3½ h. Remove the cooling water from condenser *A* and distill the vapours into the standard acid trap until the pot temperature reaches 175°C. Adjust the heating rate so that this temperature is attained in 1½ to 2½ h. Rinse condenser *B* and the connecting tube with methanol followed by distilled water, adding the rinsings to the beaker trap. Titrate the contents of the beaker with standard sodium hydroxide solution using a *p*H meter with glass-calomel electrodes and determine the end point by a difference method rather than graphically.

A-1.4 Calculation

$$\text{Diuron content, percent by mass} = \frac{23.31\times(V_1N_1 - V_2N_2)}{M}$$

where

V_1 = volume, in ml, of standard sulphuric acid;

N_1 = normality of standard sulphuric acid;

V_2 = volume, in ml, of standard sodium hydroxide solution;

N_2 = normality of standard sodium hydroxide solution; and

M = mass, in g, of sample taken for test.

A-2 AROMATIC AMINE METHOD

A-2.1 Apparatus

A-2.1.1 *pH Millivolt Meter*

A-2.1.2 *Electrodes*

A-2.1.3 *Magnetic Stirrer and Stirring Bar*

A-2.2 Reagents

A-2.2.1 *Phosphoric Acid* — 85 percent

A-2.2.2 *Chloroform*

A-2.2.3 *Sodium Hydroxide Solution* — 20 percent

A-2.2.4 *Phenolphthalein Indicator Solution*

A-2.2.5 *Glacial Acetic Acid*

A-2.2.6 *Standard Perchloric Acid in Acetic Acid* — 0.1 N, standardized. Mix 8.5 ml of 72 percent perchloric acid with 300 ml of glacial acetic acid and 20 ml of acetic anhydride. Dilute to 1 litre with glacial acetic acid and allow to stand overnight to permit complete reaction of the acetic anhydride with the water present. Standardize by titrating against primary standard potassium hydrogen phthalate, dissolved in 50 ml of glacial acetic acid and 25 ml of chloroform.

A-2.3 Procedure

A-2.3.1 Weigh sufficient sample to contain 1.0-1.5 g of active ingredient into a 100 ml flask, add 10 ml of phosphoric acid, connect a condenser to the flask and bring to a gentle boil (*see* Fig 1). Continue to reflux for 3 hrs. Cool and dilute to about 25 ml with distilled water.

A-2.3.2 Transfer the diluted hydrolysate into a 500 ml separating funnel containing 200 ml of shaved ice. Add about 25 ml of ice water, a drop of phenolphthalein indicator, and neutralize by adding cold sodium hydroxide solution in small increments. Add an additional 10 ml of sodium hydroxide solution, and extract this cold alkaline solution with one 25 ml portion of chloroform, followed by four 15 ml portions. Collect the chloroform phases in a 100 ml volumetric flask and dilute to volume. Pipette 25 ml of this solution into 50 ml glacial acetic acid contained in a 125 ml beaker. Add a magnetic stirring bar and place the beaker on stirrer platform. Immerse the electrodes, titrate with standard perchloric acid. Determine the end point graphically or by calculation.

A-2.4 Calculation

$$\text{Diuron content, percent by mass} = \frac{23.31\times(V\times N)\times 4}{M}$$

where

V = volume, in ml, of standard perchloric acid;

N = normality of standard perchloric acid; and

M = mass, in g, of sample taken for the test.

A-3 HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD

A-3.1 Principle

Diuron content is determined by high performance liquid chromatography (HPLC) equipped with UV-VIS detector using internal standard technique.

A-3.2 Apparatus

A-3.2.1 A high performance liquid chromatography system equipped with ultraviolet detector, and a printer-plotter-*cum*-integrator or PC based data system is used for this determination. The suggested operative parameters are as follows, but can be changed if necessary, provided standardization is done.

Column	:	RP C-18, 250 mm length × 4.6 mm internal diameter
Temperature	:	Ambient
Mobile phase	:	Acetonitrile : Water (60 : 40, *v/v*)
Flow rate	:	1.5 ml/ min
Injection volume	:	20 µl
Detector	:	Ultraviolet detector (260 nm)
Detector sensitivity	:	0.2 AUFS

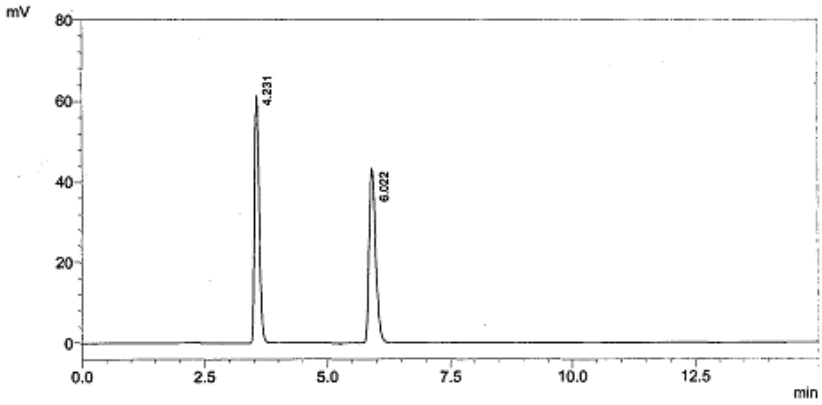


FIG. 2 A TYPICAL CHROMATOGRAM

A typical HPLC chromatogram of diuron technical with internal standard is given in Fig. 2.

A-3.2.2 *Analytical Balance*

A-3.2.3 *Microlitre Syringe*, 25 µl capacity.

A-3.2.4 *Standard Glassware*

A-3.3 Reagents

A-3.3.1 *Acetonitrile* — AR grade, or equivalent.

A-3.3.2 *Water* — AR grade, or equivalent.

A-3.3.3 *Diethyl phthalate (Internal Standard)* — of known purity.

A-3.3.4 *Diuron Reference Standard* — of known purity.

A-3.4 Procedure

A-3.4.1 *Preparation of Internal Standard Solution*

Weigh about 15.4 g of diethyl phthalate in to a 100 ml volumetric flask. Dissolve and make up the volume up to the mark with acetonitrile. Shake well to homogenize.

A-3.4.2 *Preparation of Reference Standard Solution*

Weigh accurately about 75 mg of the diuron reference standard of known purity in to a 100 ml volumetric flask. Dissolve in acetonitrile and add 5 ml of the internal standard solution. Make up the volume up to the mark with acetonitrile.

A-3.4.3 *Preparation of Sample Solution*

Weigh accurately (to the nearest 10 mg), a quantity of a sample so as to contain 75 mg of the active ingredient (diuron) in to a 100 ml volumetric flask. Dissolve in acetonitrile and add 5 ml of the internal standard solution. Make up the volume up to the mark with acetonitrile.

A-3.5 Estimation

A-3.5.1 Inject 20 µl of standard solution, until the area quotients of internal standard/standard of two successive chromatograms do not deviate from each other by more than 2 percent. Then use the following injection sequence:

$$\ldots\ldots CS_1CS_2CS_3\ldots\ldots$$

where

C = standard solution; and

S_1, S_2 = sample solutions (1, 2……., n).

From the chromatograms of the standard solution, and sample solutions, measure the peak areas of the active ingredient and the internal standard, and calculate the percentage of the active ingredient as given in **A-3.6**.

A-3.6 Retention Time (Guide Values)

Diuron	:	4.2 min (approximately)
Diethyl phthalate (Internal Standard)	:	6.0 min (approximately)

A-3.7 Calculation

$$\text{Diuron content, percent by mass} = \frac{M_1 \times A_2 \times A_3}{M_2 \times A_1 \times A_4} \times P$$

M_1 = mass in g of reference standard diuron;

M_2 = mass in g of diuron sample taken for the test;

A_1 = peak area of diuron in the reference standard solution;

A_2 = peak area of diuron in the sample solution;

A_3 = peak area of internal standard in the reference standard solution;

A_4 = peak area of internal standard in the sample solution; and

P = percent purity of diuron reference standard.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: FAD 01 (1935).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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